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BRL

UV ABSORPTION CROSS SECTIONS OF GASEOUS DIMETHYLNITRAMINE

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OCTOBER 1991

U.S. ARMY LABORATORY COMMAND

BALLISTIC RESEARCH LABORATORY
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The ultraviolet absorption spectrum of gas phase, room temperature, dimethylnitramine is reported. The spectrum contains two distinct features whose maximums occur at 193 nm (6.4 eV) and 230 nm (5.4 eV). The oscillator strength for the transition which peaks at 230 nm is determined to be 0.14 ± 0.03. Absorption cross sections at selected wavelengths between 185 nm and 325 nm have been extracted employing Beer-Lambert's law. These results are important in interpreting studies of UV photodissociation of this molecule.

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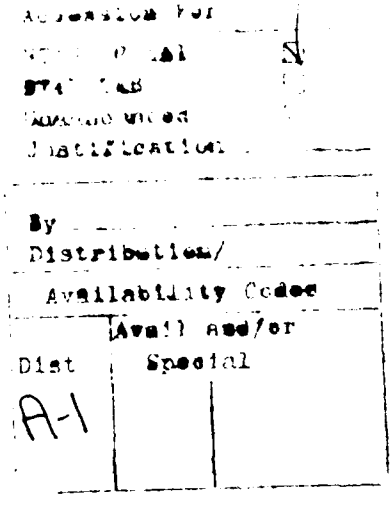

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1. INTRODUCTION

Dimethylnitramine (DMNA), $(CH_3)_2N\text{-}NO_2$, has been extensively studied as a simple analog expected to exhibit some of the reactions important in cyclic nitramine decomposition (Fluornoy 1962; Korsunski and Dubovitskii 1964, 1967; Lloyd, Umstead, and Lin 1985; McMillen et al. 1987; Nigenda, McMillen, and Golden 1989; Stewart et al. 1989; Mialocq and Stephenson 1986; Lazarou and Papagiannakopoulos 1990; Shaw and Walker 1977; Sumpter and Thompson 1987, 1988; McQuaid et al. 1991). Although many of these studies include its gas phase dissociation following excitation with UV radiation, a reference for its gas phase UV absorption cross sections has not been established. Since this is an important parameter in analyzing the results of DMNA dissociation, we report our results for room temperature DMNA. Absorption cross sections at selected wavelengths between 185 nm and 325 nm have been calculated from UV absorbance spectra employing Beer-Lambert's law.

2. EXPERIMENTAL

The purity of the DMNA sample was determined to be > 95% from mass spectrometric and IR absorption analysis. Two different systems were used to obtain absorption cross section measurements. In the first system, UV absorption spectra were recorded with a Perkin-Elmer, Lambda Array 3840, Spectrophotometer. The measurements were made in a static cell which consisted of a 10-cm long pyrex tube capped with LiF windows. A high vacuum, stainless steel, gas handling system equipped with a capacitance manometer (MKS, Model 222B) was employed for sample preparation. Upon obtaining the desired DMNA pressure, the static cell was transferred immediately to the spectrometer, and at least two absorbance spectra were recorded. It took approximately 45 s to record the range of interest, 190–325 nm. The procedure was repeated for 10 gas samples ranging in pressure from 50 to 250 mTorr. Since UV absorption will dissociate DMNA, several time scans lasting 10 min were taken to establish that DMNA depletion and concomitant buildup of photoproducts were negligible under the conditions of the experiment.

The second system used in this study utilized a large, multipurpose, stainless-steel vacuum chamber equipped with MgF_2 windows. The chamber was evacuated by a turbomolecular pump facilitating absorbance measurements under flow conditions. This

ensured against DMNA depletion and photoproduct buildup, thus providing an effective check on the results obtained with the static cell. The absorption path length for this configuration was 35 cm. The DMNA pressure for this system was also measured with a capacitance manometer (Datametrics, Type 600). The light source was an Hg pen whose spectral lines were separated with an eighth-meter monochromator (ISA Instruments, H1061). The monochromator was equipped with 1.0-mm slits producing an 8-nm FWHM band-pass filter. The light was collected with a photomultiplier tube (HAMAMATSU, R955) and the signal level measured with a digital oscilloscope (LeCroy, 9200). The linearity of the signal response with respect to input intensity was checked with neutral density filters. Two separate trials, with at least 10 signal vs. pressure readings in the range 0–250 mTorr, were taken for Hg atomic lines at 184.9 nm and 253.6 nm. No absorption was observed for DMNA vapor pressures up to 350 mTorr using Hg atomic lines at 302.1 nm and 312.6 nm.

3. RESULTS AND DISCUSSION

A typical absorption spectrum of gas phase DMNA obtained with the spectrometer is shown in Figure 1. This spectrum contains two distinct features whose maximums occur at 193 nm (6.4 eV) and 230 nm (5.4 eV). The absorption cross section (σ_λ) at selected wavelengths ($_\lambda$) was obtained by applying Beer-Lambert's law. This law may be written as follows:

$$\ln (I/I_o) = - \alpha_\lambda C \ell = - \sigma_\lambda N \ell, \qquad (1)$$

where α_λ (liter/mole cm) is the path absorption coefficient; C (mole/liter) is the concentration; ℓ (cm) the path length; σ_λ (cm^2) the absorption cross section; and N the total number of molecules per cm^3. Employing the ideal gas law, Equation 1 may be written as follows:

$$\sigma_\lambda = 2.3 \log_{10} (I_o/I) \times [n_o(P/P_o)(T_o/T)\ell]^{-1}, \qquad (2)$$

where n_o (2.6872×10^{19} part. cm^{-3}) is Loschmidt's number; P_o (760 Torr) and T_o (273 K) are standard pressure and temperature; and $\log_{10} (I_o/I)$ corresponds to the observed optical density. Figure 1 also shows the discrete values obtained at 184.9, 253.6, 302.6, and 312.6 nm using the flow cell/Hg lamp setup and 222, 226, 248, and 266 nm using the static

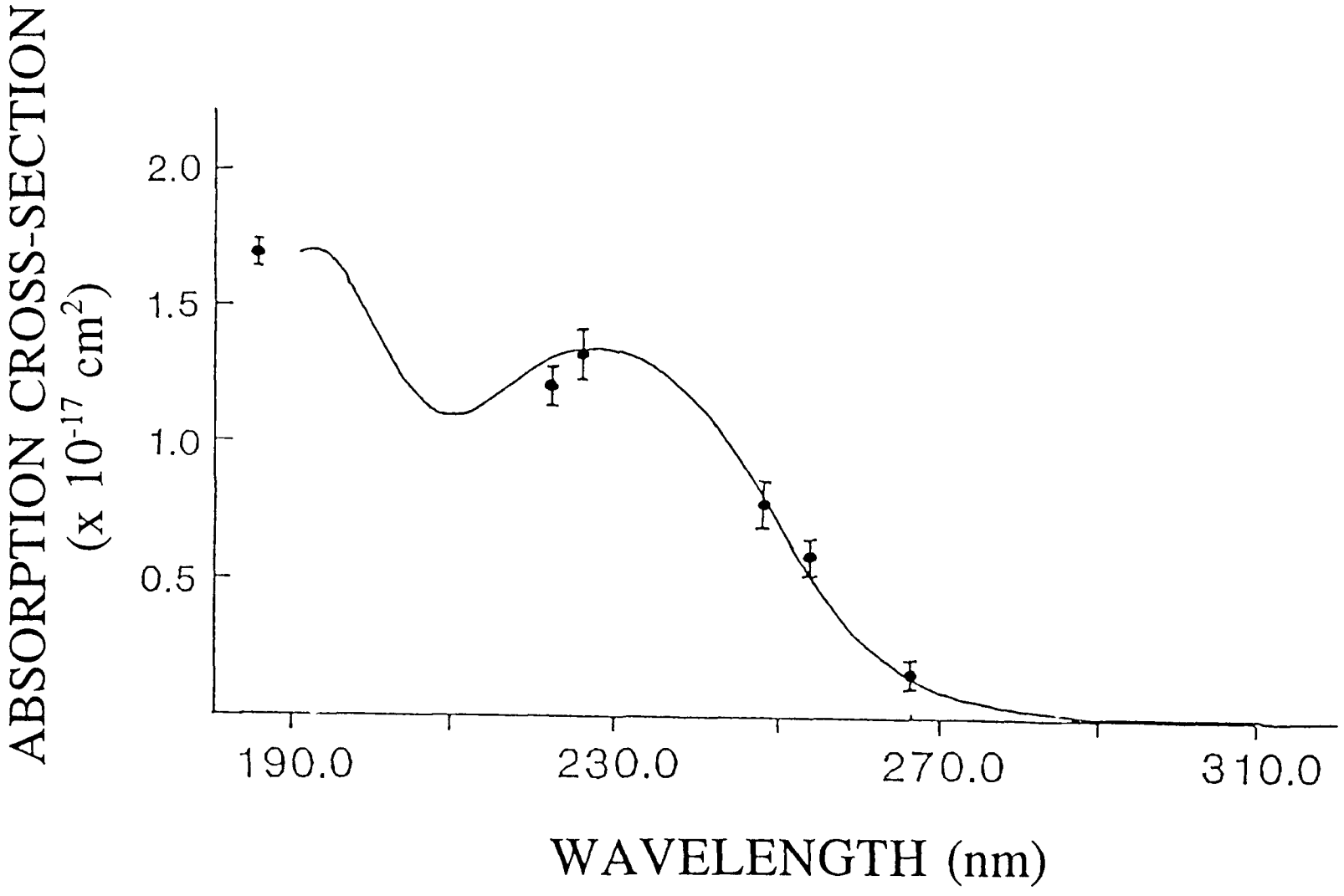

Figure 1. The Ultraviolet Absorption Spectrum of Gas Phase Dimethylnitramine.

cell/spectrometer system. The absorption cross sections obtained from the two techniques are in good agreement. Presented in Table 1 are the values obtained at common UV photolysis laser wavelengths. These results should facilitate future characterization of DMNA photolytic processes following UV excitation.

Table 1. Gaseous Dimethylnitramine Absorption Cross Sections for Selected Wavelengths

λ (nm)	Common Laser Photolysis Source	σ (cm^2)	Standard Deviation (%)
184.9		1.7×10^{-17}	3
193	Excimer (ArF)	1.7×10^{-17}[a]	—
222	Excimer (KrCl)	1.2×10^{-17}	6
248	Excimer (KrF)	7.6×10^{-18}	12
253.6		5.9×10^{-18}	3
266	Nd:YAG (Quadrupled)	1.5×10^{-18}	12
302.1		$<1 \times 10^{-19}$	—
308	Excimer (XeCl)	$<1 \times 10^{-19}$	—
312.6		$<1 \times 10^{-19}$	—

[a]Nominal value based on limited data set.

The oscillator strength for the transition which peaks at 230 nm was determined to be 0.13 ± 0.03 in good agreement with the experimentally obtained solution phase value of 0.15 reported by Stals, Barraclough, and Buchanan (1969). Semiempirical calculations predict an absorption band which peaks at 6.42 eV due to a ($^1A_1 \leftarrow {}^1A_1$) transition involving ($[n\pi\sigma \rightarrow \sigma^*_{CNNC}] + [\pi \rightarrow \pi^*]$) configurations (Stals, Barraclough, and Buchanan (1969). This value is in excellent agreement with our experimentally observed value of 6.4 eV. The semiempirical calculations also predict transitions at 5.21 eV ($^1A_1 \leftarrow {}^1A_1$) and 5.17 eV ($^1B_2 \leftarrow {}^1A_1$) attributed to ($\sigma_{CNC} \rightarrow \sigma^*_{CNC}$) and ($[\pi \rightarrow \pi^*] + [n\pi\sigma \rightarrow \sigma^*_{CNC,NO2}]$) configurations, respectively. These transitions are, however, lower in energy than our observed band at 5.4 eV. Also, the sum of the theoretically calculated oscillator strengths for these transitions (0.22) is significantly higher than our experimentally determined value of 0.14 ± 0.03. *Ab initio* calculations aimed at improving the theoretical basis for understanding the electronic transitions of this molecule are currently being considered.

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